

Contents lists available at ScienceDirect

Journal of Hazardous Materials



journal homepage: www.elsevier.com/locate/jhazmat

Removal of benzene under acidic conditions in a controlled Trickle Bed Air Biofilter

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ARTICLE INFO

Article history: Received 25 May 2010 Received in revised form 21 July 2010 Accepted 12 August 2010

Keywords: Biofiltration Biodegradation Empty Bed Residence Time (EBRT) Fungi Trickle Bed Air Biofilter (TBAB) VOCs

ABSTRACT

Trickle Bed Air Biofilters (TBABs) are considered to be economical and environmental-friendly for treatment of Volatile Organic Compounds (VOCs). Hydrophilic VOCs are easily degradable while hydrophobic ones pose a great challenge for adequate treatment due to the transfer of the VOC to the liquid phase. In this study the utilization of acidic pH is proposed for the treatment of benzene vapors. The acidic pH would encourage the growth of fungi as the main consortium. A TBAB operated at pH 4 was used for the treatment of an air stream contaminated with benzene under different loading rates ranging from 37 to 76.8 g/(m³ h). The purpose of introducing fungi was to compare the performance with traditional TBAB operating under neutral pH in order to assess the biodegradation of benzene in mixtures with other compounds favoring acidic conditions. The experimental plan was designed to assess long-term performance with emphasis based on different benzene loading rates, removal efficiency with TBAB depth, and carbon mass balance closure. At benzene loading rate of $64 \text{ g/}(\text{m}^3 \text{ h})$, the removal efficiency was 90%. At the maximum loading rate of 77 g/(m³ h), the removal efficiency was 75% marking the maximum elimination capacity for the TBAB. It is worthwhile to note that benzene appears in mixtures with n-hexane and toluene, which are reported to be better degraded under such conditions.

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1. Introduction

Benzene is a hazardous air pollution and is defined as a 'toxic air pollutant' by the Clean Air Act [1]. It is classified carcinogenic by the US Department of Health [2]. Benzene could be found in gasoline and is targeted to be lowered by 0.62% in 2011 [3]. It is used as a raw material for the synthesis of various materials including styrene, cyclohexane, aniline, phenol and alkyl benzenes [4]. OSHA set an action level of 0.5 gm^{-3} . This is the maximum airborne benzene concentration calculated as an 8-h time-weighted average [5].

The best available technology for the treatment of dilute volatile organic compounds (VOCs) is biological systems, specifically biofiltration [6]. Biological systems provide an environmentally friendly and low-cost alternative as compared to other methods such as incineration, catalytic oxidation, and adsorption. They work best for the treatment of large volumes of off-gases, which contain low concentrations of biological treatment is that it does not produce secondary effluent. Another advantage is low demand for

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supplementary material addition while in operation. Biofilters are biological systems that are promising [6]. Biofilters are packed columns with biologically active materials such as immobilized cells and compost or inorganic or polymeric media on which immobilized microbial mass is attached [7]. It was proven by several studies that biofilters are able to successfully degrade several compounds like benzene, toluene and p-xylene [9–11].

The main challenges facing biofiltration and any other biological system are the erratic changes in loadings. The changing flow rate and corresponding change in concentration of different VOCs are harmful for the micro-organisms working in the bed. The changing composition of the VOCs plays a role in hurting the removal efficiency [12]. In addition, stopping the flow during weekends or holidays might cause micro-organisms to die because of lack of food needed for growth. The microbial activity in the biofilter could decrease during non-use periods [13]. On the other hand, adequate performance could be maintained after short-term shut downs [14] but it could be significantly impacted for periods more than 2 days [15]. In addition, biofilters were proven to be a very good option for hydrophilic compounds but it becomes more reluctant when the VOC to be treated is less soluble [16].

According to Van Groenestein et al. [17], replacing the working consortium in a biofilter from Bacteria to fungi has the following advantages: (1) fungi are more resistant to acidification and dry-

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^{0304-3894/\$ -} see front matter © 2010 Elsevier B.V. All rights reserved. doi:10.1016/j.jhazmat.2010.08.042

ing out, which is a major advantage of the natural media biofilters but does not necessary count as an advantage in Trickle Air Bed Biofilters (TBABs) and (2) the aerial mycelia of fungi form a larger surface area in the gas phase than bacterial biofilms, which may facilitate the uptake of hydrophobic volatile compounds overtaking the rate limiting step. In the same study, fungi were utilized in the operation of a traditional biofilter for the treatment of toluene. Fungi were proven to be a better option for insoluble compounds like n-hexane [18]. In a later study of Kennes and Viega [19], the degradation of alkyl benzenes in a TBAB was performed using new isolated fungal strains.

The experimental plan was, therefore, designed to assess the long-term performance of benzene-fed Trickle Bed Air Biofilter (TBAB) under acidic conditions to promote fungi growth. Special emphasis were put on the following operational parameters: (1) benzene loading under increasing loading rates exceeding those reported in literature, (2) removal efficiency of the TBAB with depth under steady state conditions for evaluating the kinetic rate constant, and finally (3) volatile suspended solids and carbon mass balance closure.

2 Experimental

2.1. Trickle Bed Air Biofilter

Liquid benzene used in this study was obtained from Fisher Scientific (Fair Lawn, N.J) with 99% purity. The Henry's Law Constant (HLC) for benzene at 25° C is 5.42×10^{-3} atm m³/mol [20]. The TBAB was loaded with pellets used in a previous run where benzene has been used as the sole VOC contaminant [21]. It was found that no acclimation period was necessary. The TBAB consists of seven cylindrical glass sections with an internal diameter of 7.6 cm and a total length of 130 cm. It is packed with diatomaceous earth pelletized biological support media to a depth of about 60 cm (Celite 6 mm R-635 Bio-Catalyst Carrier; Celite Corp., Lompoc, CA). The TBAB ran at a constant operating temperature of 25 °C and operated in a cocurrent gas and liquid downward flow mode. A schematic of the TBAB setup can be found elsewhere [22].

The air flow was set up at a rate of 1.36 L/min with a corresponding EBRT of 120 s. Benzene was injected as liquid via a syringe pump and vaporized into the air stream. Buffered nutrient solution was supplied intermittently to the TBAB at a rate of 2 L/day. The composition of the nutrient solution is similar to that reported elsewhere [23]. The nutrient solution consists of essential inorganic salts and vitamins necessary to grow micro-organisms: B⁺³, Ca⁺², Cl⁻, Co⁺², CU⁺², Fe⁺³, K⁺, Mg⁺², Mn⁺², Mo⁺⁶, NH₄⁻, Na⁺, SO₄⁻², Zn⁺², p-aminobenzoic acid, biotin, cyanocobalamin, folic acid, nicotinic acid, panothenic acid, pyriodoxine hydrochloride, riboflavin, thiamin hydrochloride and thioctic acid concentration. The composition of the various components in the feed is provided in a previous publication [24]. In addition, a nutrient spike solution (2 M NaNO₃ and 0.22 M NaH₂PO₄·H₂O) was added to the feed solution so that the COD-to-nitrogen-to-phosphorus ratio was 200:4:1. NO₃ was used as the sole source of nutrient nitrogen because it was found to be effective in reducing the observed biomass yield and provided better biofilter performance as compared to NH₃ [25]. The nutrient feed was buffered at pH 4 using sodium formate. The growth of fungi within the TBAB bed was examined and identified using dichloran rose bengal chloramphenicol [26]. The average number of colonies-forming units (CFU) was determined to be 167,000 CFU/100 mL in the effluent nutrient solution. The method favors the growth of fungi by providing nitrogen, vitamins, minerals and carbohydrate, while suppressing the growth of bacteria.

The different experimental procedures that were applied to the TBAB for examining the effects on its performance are disTable 1

Operating condition for the TBAB degrading benzene under pH 4.

Experimental conditions	Phases of operation			
	Ι	II	III	IV
Inlet concentration (ppmv)	355	510	666	800
Loading rate (g/m ³ h)	34.1	48.8	64.0	76.8
(kg COD/m ³ day)	(2.51)	(3.60)	(4.72)	(5.66)
Days of operation	1-23	24-44	45-70	71-89
Average removal efficiency (%)	90.5	87.2	89.7	75.7
Standard deviation (%)	4.1	8.9	16.3	1.7
Elimination capacity (g/m ³ h)	31.3	43.2	58.2	58.8
(kg COD/m ³ day)	(2.3)	(3.2)	(10.5)	(4.3)

played in Table 1. The TBAB started initially at an influent concentration of 355 ppmv benzene which corresponds to loading rate of 34.1 g/(m^3 h) and an intermittent nutrient flow of $17.1 \text{ mmol } \text{NO}_3^-\text{N/day}$. The removal efficiency is calculated as: RE = $(C_{\text{in}} - C_{\text{out}})/C_{\text{in}}$, where RE is the removal efficiency, C_{in} is the influent concentration and C_{out} is the effluent concentration. Loading rate and elimination capacity are calculated as: LR = $C_{\text{in}} \times Q/V$ and EC = $(C_{\text{in}} - C_{\text{out}}) \times Q/V$, where Q is the air flow rate (1.34 L/min) and V is the bed volume (2.7 L).

2.2. Analytical methods

Gas phase samples were taken with gas-tight syringes by lowbleed and high-puncture-tolerance silicone gas chromatograph (GC) septa installed in the sampling ports. Benzene samples were immediately analyzed by using GC (Agilent 6890 Series, Foster City, CA) equipped with flame ionization detector and 30-m length, 0.25mm I.D., 0.25-µm film thickness narrow bore column (DB 624, J and WK Scientific, Folsom, CA). The GC oven was programmed isothermal at $120 \circ C$. The carrier gas (N₂) flowrate was set at 8 mL/min. The flame ionization detector was used with N₂ make-up gas at a flowrate of 20 mL/min, a fuel gas flow (H₂) of 30 mL/min, and an oxidizing gas flow (air) of 300 mL/min. The detector temperature was 250 °C. Retention time of 0.53 min for benzene was obtained under conditions used. The detection limit for benzene was 2 ppmv. Carbon dioxide samples were taken by using gas-tight syringes as well through sampling ports in the TBAB. A GC (HP 5890, Series II, Hewlett-Packard, Palo Alto, CA) equipped with a thermal conductivity detector was used for determining the CO₂ concentrations in the effluent gas phase. The detection limit was 0.001 vol% CO₂. Detailed description of the analytical method is provided elsewhere ([22] and [27]). The liquid phase measurements that were performed include: influent and effluent concentrations of nitrate, total carbon (TC), inorganic carbon (IC), and volatile suspended solids (VSS). Nitrate concentrations in the influent and effluent feed were determined by measuring UV absorption at wavelength of 220 nm using a Shimadzu UVmini 1240 UV-Vis spectrophotometer (Shimadzu Corp., Tokyo, Japan). Total carbon (TC) and inorganic carbon (IC) contents of the aqueous samples were determined by using a Shimadzu TOC 5000 analyzer (Shimadzu Corp., Tokyo, Japan). The VSS analysis was carried out according to Standard Methods 2540G [26].

The TBAB was operated using flow switching technique. This is performed by switching the gas flow direction weekly. This means that the gas flow would go for one week co-current with the nutrient flow downwards and then the following week would be switched upwards to counter-current the nutrient flow. Flow switching was applied in addition to stagnation as means of biomass control. Stagnation strategy is stopping all flows (VOC, nutrient, and air) passing through the TBAB which took place 2 days per week for a period of three weeks at each loading rate. This technique was chosen because it was previously reported to be a superior biomass control strat-



Fig. 1. TBAB performance under continuous loading conditions.

egy for benzene biodegradation in TBAB under neutral pH [21].

3. Results and discussion

3.1. TBAB performance

The TBAB's influent and effluent concentrations in ppmv together with the removal efficiency in percent for every day of operation at each loading rate are illustrated in Fig. 1. It is worth mentioning that during stagnation no samples were taken from the TBAB. The days of operation displayed in the plot only reflect analyzed samples during the feed of benzene to the TBAB. In Fig. 1 the day 1 of operation was the actual first day of operation as the TBAB was running under stable condition and did not require acclimation to the benzene feed. It is worthwhile to note that the TBAB was used previously for benzene feed at neutral pH. It could be seen from Fig. 1 that the effluent concentration is generally low. It was only high on the day following the stagnation period (2 days off flow of VOC, nutrient, and air). This behavior took on average a period of 1 day at lower concentrations to 2 days at the higher end for the TBAB to stabilize and achieve adequate performance.

The different stages of the TBAB operation are elaborated in details in Table 1. The TBAB was started up at 355 ppmv benzene influent concentration, 2-min EBRT and loading rate $34 g/(m^3 h)$. The overall removal efficiency had reached 90% and was stable at this level for 23 days. On day 24, the influent concentration was then increased to 510 ppmv at 48.7 g/(m^3 h) . At this concentration level, the removal efficiency dropped slightly to 87%. On day 45, the influent concentration was raised again to 666 ppmv. The removal efficiency remained stable at 89.7%. There was a slight increase on the average of the removal efficiency, however, the standard deviation doubled to 16%. The increase in the standard deviation is due to the high peaks that occurred immediately after stagnation. During the course of the week the removal efficiency stabilized to a better performance. On day 71, the maximum concentration level applied to the TBAB was 800 ppmv. At this concentration level, the performance of TBAB decreased and the average removal efficiency went down to 76%. On the other hand, the standard deviation was very low (see Table 1). This was due to the stable performance attained. The only peak of high effluent concentration level was noticed on the first day immediately after the influent concentration was raised.

Fig. 2 shows the elimination capacity vs. loading rates for acidic and neutral pH. The acidic pH 4 is the main focus of the study, while the neutral pH was studied in a previous study [21]. Both performances were displayed for comparison reasons. The plot



Fig. 2. Elimination capacity at different loading rates. *Performance at neutral pH is displayed for comparison reasons [19].

includes a 100% removal efficiency line. For acidic pH, the elimination capacity was increasing steadily with increase of loading rate up to $64 \text{ g}/(\text{m}^3 \text{ h})$. After this level, there was no increase in the elimination capacity. The maximum elimination capacity attained was $58 \text{ g}/(\text{m}^3 \text{ h})$. The elimination capacity at neutral pH was slightly higher than those at acidic pH. The maximum elimination capacity at neutral pH was $65 \text{ gg}/(\text{m}^3 \text{ h})$. It is worth mentioning that the difference between both operating conditions is not significant. Operating at acidic pH on the other hand, ensures that benzene could be degraded in fungi environment. This study opens possibilities of degrading benzene in gaseous mixtures containing hydrophobic compounds that are more favorable to be degraded in fungi environment [28].

In recent studies, the treatment of benzene in biofilters was studied in mixtures. Typical mixtures of benzene, toluene, and xylene [29] or benzene, toluene, ethyl benzene and xylene (BTEX) [23,30,31] were studied. The maximum elimination capacity for benzene found in the literature as reported is $34 g/(m^3 h)$ by Lu et al. [31]. This value is by far less than the value reported by this study 58 g/(m³ h). Benzene was studied in mixture with toluene and high elimination capacities were reported but short operation study time might be behind the high elimination capacities [32]. In another study benzene was mixed with monochlorobenzene but very low loading rates of benzene of $2g/(m^3 h)$ were applied [33]. Benzene, being a single substrate source, was studied only in traditional biofilters [30,34–36]. Mathur et al. [36] found a maximum elimination capacity of 45 g/(m³ h) running only at 65% removal efficiency. These previous studies clearly show the enhancements obtained in this study in a TBAB over traditional biofilters.

3.2. Carbon mass balance

In Fig. 3, the cumulative CO_2 equivalent of benzene in moles at the inlet was compared to the same equivalent at the effluent of the TBAB. The influent cumulative CO_2 consists of two main components: influent gaseous concentration and influent aqueous inorganic and organic carbon. The effluent CO_2 equivalent includes the effluent aqueous inorganic and organic carbon, effluent VSS, gaseous CO_2 and effluent benzene concentration. Fig. 3 indicates that the carbon recovery was 89% with a standard deviation of 3%. It is assumed that the loss in carbon between the influent and effluent was retained as biomass within the TBAB. The hypothesis was assessed by comparing the loss in carbon to the biomass amount accumulated in the TBAB which was calculated using the



Fig. 3. Cumulative carbon input/output as CO₂ equivalent in mol.

daily nitrate consumption for all loading rates. The typical cellular composition for heterogeneous microorganism is represented as $C_9H_{15}O_5N$ [37,38] was used as the basis for relating the nitrate consumed in building up new biomass in order to estimate the amount of biomass retained within the TBAB. Results of the *t*-test indicate that the carbon losses due to biomass accumulated in the TBAB was highly significant with a *p*-value < 0.05. Therefore, it is assumed that the difference in carbon between the influent and effluent carbon is retained within the bed for building up new biomass.

It is worthwhile noting that the main key contributors to the carbon cycle are the gas phase concentrations, namely, influent and effluent benzene concentration, and effluent gaseous carbon dioxide. The contribution of the different carbon components is provided in Fig. 4. All the different components contributing to the carbon balance are represented as daily measurements with exception to the effluent carbon equivalence of benzene and the gaseous CO₂ carbon equivalent which are represented as box plot for each concentration level. The box plot summary indicates the 25th and 75th percentiles by the borders of the box, the median by the line within the box, and the 90th and 10th percentiles by the error bars. For the box plot representing the effluent gaseous CO₂ at the highest loading rate in Fig. 4, the 25th, median, 75th and 90th percentiles coincide on the top line, while the lower line represents the 10th percentile. The carbon share in the liquid phase due to the amount calculated from VSS, influent and effluent organic carbons in the aqueous phase can be considered to be negligible since the total aqueous carbon did not exceed 5% of the total carbon in the system (gaseous and liquid phases). Although the carbon in the influent and effluent liquid value if considered separately is high but the



Fig. 4. Sources of carbon in the TBAB.

difference between influent and effluent is negligible. Flow switching did not cause loss of benzene in the liquid phase when the gas and liquid phases are run counter-currently.

3.3. Kinetics of TBAB performance

The removal performance at different bed depth was measured weekly. This was performed after 1 day following stagnation at the sampling ports, located along the depth at 7.6, 23, 38, 53 and 60 cm measured from media top. These data were used to develop the pseudo first order reaction rate constant as a function of time. In order to avoid the misinterpretation of data, the kinetic analysis was conducted using the data from sampling ports within the media as there is a possibility of biodegradation on the top portion of the TBAB above the media or at the bottom disengagement chamber used for separation of liquid and gas effluents. The reaction rate constant is calculated under the assumption of pseudo first order reaction occurring in a plug flow reactor [25,39]. Sampling data at every concentration (minimum of 3 dataset) were fit to first order kinetics model with the independent variable, time (s), and the dependant variable, $log(C/C_0)$, where C is the effluent concentration and C_0 is the influent concentration as measured at the first port. The coefficient of regression R^2 for the linear fit has a minimum of 0.92 for all the cases studied. The average reaction rate constant was 0.0096, 0.0112 and $0.0189 \, \text{s}^{-1}$ for 350, 510 and 666 ppmv, respectively. In neutral pH under the same condition similar values were reported to be 0.0111, 0.0109 and 0.0024 s⁻¹, respectively. Sorial et al. [24] reported comparable values for biodegradation of benzene in BTEX mixture in a TBAB. The reported values were in the range of 0.0196 s⁻¹ at a loading rate of 14 g/(m^3 h) to 0.0448 s⁻¹ at a loading rate of $42 g/(m^3 h)$. In the same study similar toluene values were found in the BTEX mixture. They ranged from 0.0296 to $0.0195 \,\mathrm{s}^{-1}$ at the same benzene loading rate. In another study Kim et al. [40] reported the same trend for toluene ranging from 0.01 s^{-1} at a loading rate of 10 g/(m^3 h) to 0.04 s^{-1} at a loading rate of $49 g/(m^3 h)$.

Further analysis was conducted by fitting the daily performance data to a plug flow model that was developed in an earlier study [21] in order to estimate the TBAB reaction kinetics during the different operation strategies. According to the model the first order pseudo reaction rate constants (k) were found to be 0.00959, 0.00805 and 0.00133 s⁻¹ for 350, 510 and 666 ppmv, respectively. The maximum relative difference between the k values obtained from the model and the k values obtained above is 0.1%, 28.1% and 29.6%, respectively. Thus, the model can estimate the performance of the TBAB within a reasonable accuracy for each concentration level studied.

4. Conclusions

The loading rate used is relatively high if compared to typical biofilter operations. For the lowest loading rate of 34 g/(m^3 h) , the removal efficiency was 90% and the performance was almost stable at this level up to a loading rate of $64 g/(m^3 h)$. The maximum elimination capacity was found to be 58 g/(m^3 h) . It is superior to most published literature but is slightly lower than the same setup running under neutral pH reported in our previous study. It is important to notice that for long-term stable performance of TBAB utilizing stagnation as biomass control strategy, backwashing will be required periodically to reduce effectively the excess biomass retained within the bed. An important observation noticed was the lack of acclimation period when the pH environment was changed. Such observation is vital for biofilter facilities looking for shifting from neutral to acidic pH environments due to the presence of hydrophobic contaminants that require such conditions. At the same time there is no noticeable difference in performance for contaminants that can degrade under both neutral and acidic pH.

Operating the TBAB under acidic pH without major sacrifice in performance gives the opportunity for utilization of TBABs for benzene in mixtures under these conditions. Other compounds that would typically appear together with benzene in mixtures were proven to better biodegrade under acidic environment e.g. n-hexane. In addition, toluene, which behaves similarly in biodegradation as benzene, could be a good candidate for biodegradation under acidic environment. Toluene appears with benzene in two important mixtures namely, BTX and BTEX.

Acknowledgements

The authors would like to thank the financial support from National Science Foundation under award # CBET 0852803. The finding and conclusions expressed in this publication are solely those of the authors and do not necessary reflect the views of the Foundation.

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